

BEST AVAILABLE COPY
PATENT ABSTRACTS OF JAPAN

(11)Publication number : 05-194629

(43)Date of publication of application : 03.08.1993

(51)Int.Cl.

C08F 8/04

(21)Application number : 04-025974

(71)Applicant : TOONETSUKUSU KK

(22)Date of filing : 17.01.1992

(72)Inventor : OZAWA MASAO
FUJIWARA HIDEKI
NAKANO YOSHIO
MATSUZAKI KIMISHIGE

(54) HYDROGENATED PETROLEUM RESIN

(57)Abstract:

PURPOSE: To provide a hydrogenated petroleum resin suitable for employment as an adhesive-imparting agent for white (pale colored) hot-melt adhesive compositions and having excellent compatibility, initial adhesive force (tack), adhesive force and holding force.

CONSTITUTION: A resin obtained by subjecting (A) 50-90wt.% of a cyclopentadienic component, (B) a vinyl-substituted aromatic component and (C) an indenic aromatic component in a total amount of 10-50wt.%, and (C) a raw material in a B/C weight ratio of (0.6-4.0)/1 to a ternary copolymerization is hydrogenated to obtain the hydrogenated petroleum resin having specific properties.

LEGAL STATUS

[Date of request for examination] 12.01.1999

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number] 3173841

[Date of registration] 30.03.2001

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

* NOTICES *

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] Cyclopentadiene system component (A) Hydrogenation petroleum resin which has 2 - 25 % of the weight of residual nucleus contents, 15 or less cg/g of iodine numbers, and 80-150 degrees C of softening temperatures which consisted of a vinyl permutation aromatic series component (B) and a total of 10 - 50% of the weight of an indene system aromatic series component (C) 50 to 90% of the weight, and hydrogenated the resin obtained by carrying out ternary polymerization of the raw material whose weight ratios (B/C) of a component (B) and a component (C) are 0.6-4.0.

[Translation done.]

* NOTICES *

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to hydrogenation petroleum resin with a hue and heat-resistant stability especially useful as a tackifier of a white (light color) system hot melt binder constituent good in more detail about new hydrogenation petroleum resin.

[0002]

[Description of the Prior Art] As thermoplastic elastomer used as a base material of a white (light color) system hot melt binder, white system synthetic rubber, such as styrene system block-copolymer elastomers, such as SBS, SIS, and SEBS, and EVA, is mainly used from the point of quality, thermal resistance, and weatherability in recent years. Development of a useful tackifier is demanded to these elastomers.

[0003] The physical properties demanded as a tackifier of such a white (light color) system hot melt binder are adhesion properties, such as a hue, thermal resistance, weatherability, compatibility with a base elastomer, initial adhesion (tack) when considering as a hot melt binder further, adhesion, and holding power.

[0004] As this tackifier, natural resin, such as terpene system resin and rosin system resin, has been conventionally used widely from that outstanding adhesive effectiveness. However, since it is a natural product, it is unstable in respect of a price and supply. It is in the inclination to be used replacing petroleum resin with natural system resin in recent years, and hydrogenation petroleum resin is especially used from the field of a hue and heat-resistant stability.

[0005] Although the hydrogenation petroleum resin by which current use is carried out is the hydrogenation object of cyclopentadiene system resin and aromatic series petroleum resin, these run short of compatibility or adhesive ability with a base elastomer. Moreover, the new hydrogenation petroleum resin by copolymerization is known for the purpose of amelioration of these faults. For example, the hydrogenation hydrocarbon resin guided to JP,61-1442,B from 20 - 80 % of the weight of cyclopentadiene system components by which hydrogenation was carried out, and 20 - 50 % of the weight of mono-vinyl permutation aromatic series components by which hydrogenation was carried out is indicated. Although other components may live together, it is specifically unstated.

[0006] What carried out partial hydrogenation of the resin which copolymerized and obtained the cyclopentadiene system compound 70 - 95 weight sections, and the indene system compound 30 - 5 weight sections to JP,61-8849,B is indicated. Aromatic series C9 which carries out a byproduction by naphtha cracking as an indene system compound It is supposed that the fraction (35% or more of indene contents) by fractional distillation of a fraction can be used. In this fraction, the styrene system monomer other than an indene can be included in 20% or less of amount, therefore the ratio of styrene/indene is 0.57 or less. In the example, this ratio is 0.38. That is, there are few amounts of the styrene to an indene.

[0007] However, all the physical properties demanded as the above-mentioned tackifier were not able to be filled with such hydrogenation resin.

[0008]

[Problem(s) to be Solved by the Invention] This invention offers hydrogenation petroleum resin excellent in adhesion properties, such as initial adhesion (tuck) when it being made in view of such a trouble, improving the fault of old hydrogenation petroleum resin, and excelling in the hue and the heat-resistant stability which respond to the request of the latest commercial scene, and compatibility with a base elastomer, and considering as a hot melt binder, adhesion, and holding power.

[0009]

[Means for Solving the Problem] As a result of repeating research wholeheartedly that the above-mentioned technical problem should be solved, this invention persons Cyclopentadiene system (component A) 50-90 % of the weight, It consists of a vinyl permutation aromatic series component (B) and a total of 10 - 50% of the weight of an indene system aromatic series component (C). And hydrogenated the resin with which the weight ratio (B/C) of a component (B) and a component (C) carries out ternary polymerization of the raw material which are 0.6-4.0, and is obtained. This invention which consists of hydrogenation petroleum resin which has 2 - 25 % of the weight of residual nucleus contents, 15 or less cg/g of iodine numbers, and 80-150 degrees C of softening temperatures was completed.

[0010] That is, as a result of considering the resin design of the petroleum resin as a tackifier of a hot melt binder, this invention persons find out demonstrating the effectiveness which was excellent when the structure of resin and the relation of physical properties were as follows, and came to complete the hydrogenation petroleum resin of this invention.

[0011] Compatibility copolymerizes an aromatic series monomer 10% of the weight or more. The weight average molecular weight (Mw) of resin 5000 or more macromolecule fields Two or less % of the weight, It increases by carrying out to 1 or less % of the weight preferably. Adhesion 2.5 or less, if the ratio (Mw/Mn) of the weight average molecular weight and number average molecular weight of resin becomes 2.0 or less preferably, it will increase, and holding power is [weight average molecular weight (Mw)] preferably excellent in 2% of the weight or more of the thing for the rate of or more 3000 less than 5000 field 1% of the weight or more.

[0012] The Diels-Alder addition product of the lower order like low-grade alkylation cyclopentadienes, such as a cyclopentadiene, methylation, and an ethyl permutation, and these dimers, a trimer, and ***** is contained in the cyclopentadiene system component (A) used as a raw material by this invention.

[0013] Styrene, vinyltoluene, a divinylbenzene, alpha methyl styrene, beta-methyl styrene, etc. are contained in the vinyl permutation aromatic series component (B) similarly used as a raw material.

[0014] Similarly, low-grade alkylation indenenes, such as an indene, methylation, and an ethyl permutation, are contained in an indene system aromatic series component (C).

[0015] Next, the manufacture approach of hydrogenation petroleum resin useful as a tackifier of the white (light color) system hot melt binder of this invention is explained.

[0016] First, it consists of a vinyl permutation aromatic series component (B) and a total of 10 - 50% of the weight of an indene system aromatic series component (C) cyclopentadiene system (component A) 50-90% of the weight. The raw material prepared so that the weight ratio (B/C) of a component (B) and a component (C) might be set to 0.6-4.0 under existence of a solvent. And the reaction temperature of 240-300 degrees C, the solvent after carrying out thermal polymerization in reaction-time 30 - 300 minutes -- and unreacted or a low polymerization component is removed and resin (I) of 60-130 degrees C of softening temperatures is obtained. As a solvent used here, a pentane, a hexane, a heptane, benzene, toluene, a xylene, etc. are raised.

[0017] In addition, resin (I) can also obtain the polymerization reaction by the catalyst of the Friedel Kraft molds, such as a boron trifluoride and an aluminum chloride, at the temperature of 0-100 degrees C other than the above-mentioned thermal polymerization.

[0018] Furthermore, hydrogenation is carried out according to the approach of hydrogenating this resin (I) at the temperature of 100-400 degrees C under existence of a diluent using catalysts, such as a well-known approach, for example, nickel, palladium, cobalt, platinum, and a rhodium system. It is required to carry out hydrogenation on the occasion of hydrogenation

nearly completely [the hydrogenation rate] about the partial saturation double bond in resin, i.e., to consider as 95% or more of rates of hydrogenation of an ethylene nature partial saturation radical, and it is required about a nucleus to limit to 25 - 75% of partial hydrogenation of hydrogenation, i.e., a rate.

[0019] Hydrogenation petroleum resin of 2 - 25 % of the weight of residual aromatic series contents which have 80-150 degrees C of softening temperatures, and were measured by 15 or less cg/g of iodine numbers and C13-NMR is obtained by removing a diluent and a low-molecular-weight object after hydrogenation. The Gardner hue is one or less.

[0020] In performing the above-mentioned thermal polymerization reaction, in this invention, a raw material presentation must fulfill the conditions of this invention. That is, a component (A) is 50 - 70 % of the weight preferably 50 to 90% of the weight, and a component (B) and a component (C) are 30 - 50 % of the weight preferably a total of ten to 50% of the weight, and the weight ratio (B/C) of a component (B) and a component (C) is required for 0.6-4.0, and being the range of 1.0-3.0 preferably.

[0021] If there are few components (A) than 50 % of the weight, the softening temperature of resin (I) runs short, and if [than 90 % of the weight] more (i.e., if there is less sum total of a component (B) and a component (C) than 10 % of the weight), compatibility with a base elastomer will get worse.

[0022] Moreover, adhesion is not enough, when molecular-weight-distribution Mw/Mn becomes large, hydrogenates and uses as a hot melt binder, while the rate of the high polymerization object in resin (I) will increase and compatibility with a base elastomer will get worse, if a ratio (B/C) is larger than 4.0.

[0023] Moreover, if a ratio (B/C) is smaller than 0.6, the yield at the time of the polymerization of resin (I) falls and is uneconomical, and the holding power at the time of the rates of a moderate macromolecule field running short and using as a binder is not enough.

[0024] It is thought that the phenomenon about such molecular weight is based on the reactant difference between a component (B) and a component (C). That is, compared with an indene system aromatic series component (C), a vinyl permutation aromatic series component (B) is for reactivity to tend to generate a high polymerization object highly.

[0025] Although an example is given below and this invention is explained still more concretely, this invention is not limited to these examples. In addition, especially % in an example and the example of a comparison is weight criteria unless it refuses.

[0026]

[Examples 1-3] It taught at a rate which it considers as a dicyclopentadiene as a component (A), is considered a 1l. autoclave with styrene and a component (C) as a component (B), and shows an indene in Table 1, and thermal polymerization was carried out to the bottom of nitrogen-gas-atmosphere mind as a solvent using toluene in reaction temperature [of 275 degrees C], and reaction-time 100 minutes. After reaction termination, distillation removed the low polymerization component and a solvent and the resin (I) which has unreacted or the description shown in Table 1 were obtained.

[0027] Then, 2.0g (N-113, JGC make) of nickel system catalysts was taught to the 0.2l. autoclave, having used the cyclohexane as 80g and a catalyst for this resin (I) as 80g and a diluent, and the hydrogenation reaction was performed at G and 300 degrees C 150kg/cm² of hydrogen pressure. After reaction termination, distillation removed the diluent and hydrogenation petroleum resin was obtained. The description of hydrogenation petroleum resin was shown in Table 1.

[0028] The hydrogenation petroleum resin obtained by this invention is excellent in compatibility, and it turns out initial adhesion (tuck), adhesion, holding power, and that it excels also in economical efficiency further so that clearly from Table 1. Moreover, the hydrogenation petroleum resin of the example especially manufactured under the desirable conditions of a ratio (B/C) was very excellent in the description of all above.

[0029]

[The examples 1-5 of a comparison] Hydrogenation petroleum resin was manufactured using the raw material presentation which is under the same condition as the above-mentioned example,

however is shown in Table 1. The description of the obtained hydrogenation petroleum resin was as being shown in Table 1.

[0030] Thus, the hydrogenation petroleum resin obtained by the example of a comparison was fully able to satisfy no binder engine performance as stated above.

[0031]

[Table 1]

表 1

	実 施 例			比 較 例				
	1	2	3	1	2	3	4	5
原料組成、%								
成分 (A)	55	55	55	92	40	55	55	55
成分 (B) + (C)	45	45	45	8	60	45	45	45
(B)/(C)	2.3	4.0	0.7	2.0	0.2	0	0.5	∞
樹脂 (I) 性状								
軟化点、℃	85	87	86	86	70	84	86	83
収率、%	80	81	79	81	79	70	76	82
I ₂ 価, cg/g	215	216	208	235	192	208	205	211
芳香核含量、%	31	32	31	5.6	41	31	32	31
分子量								
Mw	930	1050	850	550	810	530	680	2100
Mw/Mn	1.97	2.13	1.88	1.94	1.82	1.65	1.76	4.11
Mw>5000、%	0.87	1.54	0.45	0.52	0.35	0.33	0.27	9.83
5000>Mw>3000、%	3.05	4.01	2.13	1.45	1.23	0.63	1.07	5.50
水添石油樹脂性状								
軟化点、℃	104	104	103	105	85	102	102	103
I ₂ 価, cg/g	7.7	8.5	8.0	5.0	9.6	9.0	8.8	7.9
芳香核含量、%	19.1	19.5	19.3	1.5	30.8	19.8	20.0	18.9
水添率、%								
エチレン性								
不飽和基	96.4	96.1	96.2	97.9	95.0	95.7	95.7	96.3
芳香核	38.4	39.1	37.7	73.2	24.9	36.1	37.5	39.0
粘着剤性能								
相溶性	◎	○	◎	×	○	◎	◎	×
接着力, g/20mm	1350	1100	1350	300	1200	1550	1400	600
保持力, Hr.	1.5	1.4	1.1	0.5	0.2	0.5	0.8	1.1

The rate of hydrogenation of an ethylene nature partial saturation radical, the rate of hydrogenation of a nucleus, compatibility, adhesive strength, and holding power show the following contents among Table 1.

[0032]

Rate (%) of hydrogenation = (aromatic series content of aromatic series content / petroleum resin before hydrogenation of 1-hydrogenation petroleum resin) x100 compatibility of rate (%) of hydrogenation = (I₂ of 1-hydrogenation petroleum resin I₂ of ** / petroleum resin before hydrogenation **) x100 nucleus of an ethylene nature partial saturation radical : EVA / petroleum resin / wax Blend with 40/40/20 (weight ratio), and an observation adhesive strength: melting sample is applied to the thickness of 0.1mm for the transparency of the mixed

liquor which fully carried out dissolution mixing on an aluminium sheet. A spreading side comrade is pasted up at 160 degrees C using a heat sealer, a measurement holding power:melting sample is applied to corrugated paper adherend at the hauling rate of per minute 30cm with a hauling testing machine, and it sticks on another corrugated paper adherend immediately. Time amount until it applies a 200g load in 45-degree-C oven and a spindle falls this test piece [0033]
[Effect of the Invention] As mentioned above, this invention is excellent in compatibility, and offers useful hydrogenation petroleum resin especially as a tackifier of initial adhesion (tuck), adhesion, holding power, and the white (light color) system hot melt binder constituent that was further excellent also in economical efficiency.

[Translation done.]

* NOTICES *

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1.This document has been translated by computer. So the translation may not reflect the original precisely.

2.*** shows the word which can not be translated.

3.In the drawings, any words are not translated.

TECHNICAL FIELD

[Industrial Application] This invention relates to hydrogenation petroleum resin with a hue and heat-resistant stability especially useful as a tackifier of a white (light color) system hot melt binder constituent good in more detail about new hydrogenation petroleum resin.

[Translation done.]

* NOTICES *

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

PRIOR ART

[Description of the Prior Art] As thermoplastic elastomer used as a base material of a white (light color) system hot melt binder, white system synthetic rubber, such as styrene system block-copolymer elastomers, such as SBS, SIS, and SEBS, and EVA, is mainly used from the point of quality, thermal resistance, and weatherability in recent years. Development of a useful tackifier is demanded to these elastomers.

[0003] The physical properties demanded as a tackifier of such a white (light color) system hot melt binder are adhesion properties, such as a hue, thermal resistance, weatherability, compatibility with a base elastomer, initial adhesion (tack) when considering as a hot melt binder further, adhesion, and holding power.

[0004] As this tackifier, natural resin, such as terpene system resin and rosin system resin, has been conventionally used widely from that outstanding adhesive grant effectiveness. However, since it is a natural product, it is unstable in respect of a price and supply. It is in the inclination to be used replacing petroleum resin with natural system resin in recent years, and hydrogenation petroleum resin is especially used from the field of a hue and heat-resistant stability.

[0005] Although the hydrogenation petroleum resin by which current use is carried out is the hydrogenation object of cyclopentadiene system resin and aromatic series petroleum resin, these run short of compatibility or adhesive ability with a base elastomer. Moreover, the new hydrogenation petroleum resin by copolymerization is known for the purpose of amelioration of these faults. For example, the hydrogenation hydrocarbon resin guided to JP,61-1442,B from 20 - 80 % of the weight of cyclopentadiene system components by which hydrogenation was carried out, and 20 - 50 % of the weight of mono-vinyl permutation aromatic series components by which hydrogenation was carried out is indicated. Although other components may live together, it is specifically unstated.

[0006] What carried out partial hydrogenation of the resin which copolymerized and obtained the cyclopentadiene system compound 70 - 95 weight sections, and the indene system compound 30 - 5 weight sections to JP,61-8849,B is indicated. Aromatic series C9 which carries out a byproduction by naphtha cracking as an indene system compound It is supposed that the fraction (35% or more of indene contents) by fractional distillation of a fraction can be used. In this fraction, the styrene system monomer other than an indene can be included in 20% or less of amount, therefore the ratio of styrene/indene is 0.57 or less. In the example, this ratio is 0.38. That is, there are few amounts of the styrene to an indene.

[0007] However, all the physical properties demanded as the above-mentioned tackifier were not able to be filled with such hydrogenation resin.

[Translation done.]

* NOTICES *

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

EFFECT OF THE INVENTION

[Effect of the Invention] As mentioned above, this invention is excellent in compatibility, and offers useful hydrogenation petroleum resin especially as a tackifier of initial adhesion (tuck), adhesion, holding power, and the white (light color) system hot melt binder constituent that was further excellent also in economical efficiency.

[Translation done.]

* NOTICES *

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.

2. **** shows the word which can not be translated.

3. In the drawings, any words are not translated.

TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] This invention offers hydrogenation petroleum resin excellent in adhesion properties, such as initial adhesion (tack) when it being made in view of such a trouble, improving the fault of old hydrogenation petroleum resin, and excelling in the hue and the heat-resistant stability which respond to the request of the latest commercial scene, and compatibility with a base elastomer, and considering as a hot melt binder, adhesion, and holding power.

[Translation done.]

* NOTICES *

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

MEANS

[Means for Solving the Problem] As a result of repeating research wholeheartedly that the above-mentioned technical problem should be solved, this invention persons Cyclopentadiene system (component A) 50-90 % of the weight, It consists of a vinyl permutation aromatic series component (B) and a total of 10 - 50% of the weight of an indene system aromatic series component (C). And hydrogenated the resin with which the weight ratio (B/C) of a component (B) and a component (C) carries out ternary polymerization of the raw material which are 0.6-4.0, and is obtained. This invention which consists of hydrogenation petroleum resin which has 2 - 25 % of the weight of residual nucleus contents, 15 or less cg/g of iodine numbers, and 80-150 degrees C of softening temperatures was completed.

[0010] That is, as a result of considering the resin design of the petroleum resin as a tackifier of a hot melt binder, this invention persons find out demonstrating the effectiveness which was excellent when the structure of resin and the relation of physical properties were as follows, and came to complete the hydrogenation petroleum resin of this invention.

[0011] Compatibility copolymerizes an aromatic series monomer 10% of the weight or more. The weight average molecular weight (Mw) of resin 5000 or more macromolecule fields Two or less % of the weight, It increases by carrying out to 1 or less % of the weight preferably. Adhesion 2.5 or less, if the ratio (Mw/Mn) of the weight average molecular weight and number average molecular weight of resin becomes 2.0 or less preferably, it will increase, and holding power is [weight average molecular weight (Mw)] preferably excellent in 2% of the weight or more of the thing for the rate of or more 3000 less than 5000 field 1% of the weight or more.

[0012] The Diels-Alder addition product of the lower order like low-grade alkylation cyclopentadienes, such as a cyclopentadiene, methylation, and an ethyl permutation, and these dimers, a trimer, and ***** is contained in the cyclopentadiene system component (A) used as a raw material by this invention.

[0013] Styrene, vinyltoluene, a divinylbenzene, alpha methyl styrene, beta-methyl styrene, etc. are contained in the vinyl permutation aromatic series component (B) similarly used as a raw material.

[0014] Similarly, low-grade alkylation indenenes, such as an indene, methylation, and an ethyl permutation, are contained in an indene system aromatic series component (C).

[0015] Next, the manufacture approach of hydrogenation petroleum resin useful as a tackifier of the white (light color) system hot melt binder of this invention is explained.

[0016] First, it consists of a vinyl permutation aromatic series component (B) and a total of 10 - 50% of the weight of an indene system aromatic series component (C) cyclopentadiene system (component A) 50-90% of the weight. The raw material prepared so that the weight ratio (B/C) of a component (B) and a component (C) might be set to 0.6-4.0 under existence of a solvent And the reaction temperature of 240-300 degrees C, the solvent after carrying out thermal polymerization in reaction-time 30 - 300 minutes -- and unreacted or a low polymerization component is removed and resin (I) of 60-130 degrees C of softening temperatures is obtained. As a solvent used here, a pentane, a hexane, a heptane, benzene, toluene, a xylene, etc. are raised.

[0017] In addition, resin (I) can also obtain the polymerization reaction by the catalyst of the

Friedel Kraft molds, such as a boron trifluoride and an aluminum chloride, at the temperature of 0–100 degrees C other than the above-mentioned thermal polymerization.

[0018] Furthermore, hydrogenation is carried out according to the approach of hydrogenating this resin (I) at the temperature of 100–400 degrees C under existence of a diluent using catalysts, such as a well-known approach, for example, nickel, palladium, cobalt, platinum, and a rhodium system. It is required to carry out hydrogenation on the occasion of hydrogenation nearly completely [the hydrogenation rate] about the partial saturation double bond in resin, i.e., to consider as 95% or more of rates of hydrogenation of an ethylene nature partial saturation radical, and it is required about a nucleus to limit to 25 – 75% of partial hydrogenation of hydrogenation, i.e., a rate.

[0019] Hydrogenation petroleum resin of 2 – 25 % of the weight of residual aromatic series contents which have 80–150 degrees C of softening temperatures, and were measured by 15 or less cg/g of iodine numbers and C13-NMR is obtained by removing a diluent and a low-molecular-weight object after hydrogenation. The Gardner hue is one or less.

[0020] In performing the above-mentioned thermal polymerization reaction, in this invention, a raw material presentation must fulfill the conditions of this invention. That is, a component (A) is 50 – 70 % of the weight preferably 50 to 90% of the weight, and a component (B) and a component (C) are 30 – 50 % of the weight preferably a total of ten to 50% of the weight, and the weight ratio (B/C) of a component (B) and a component (C) is required for 0.6–4.0, and being the range of 1.0–3.0 preferably.

[0021] If there are few components (A) than 50 % of the weight, the softening temperature of resin (I) runs short, and if [than 90 % of the weight] more (i.e., if there is less sum total of a component (B) and a component (C) than 10 % of the weight), compatibility with a base elastomer will get worse.

[0022] Moreover, adhesion is not enough, when molecular-weight-distribution Mw/Mn becomes large, hydrogenates and uses as a hot melt binder, while the rate of the high polymerization object in resin (I) will increase and compatibility with a base elastomer will get worse, if a ratio (B/C) is larger than 4.0.

[0023] Moreover, if a ratio (B/C) is smaller than 0.6, the yield at the time of the polymerization of resin (I) falls and is uneconomical, and the holding power at the time of the rates of a moderate macromolecule field running short and using as a binder is not enough.

[0024] It is thought that the phenomenon about such molecular weight is based on the reactant difference between a component (B) and a component (C). That is, compared with an indene system aromatic series component (C), a vinyl permutation aromatic series component (B) is for reactivity to tend to generate a high polymerization object highly.

[0025] Although an example is given below and this invention is explained still more concretely, this invention is not limited to these examples. In addition, especially % in an example and the example of a comparison is weight criteria unless it refuses.

[0026]

[Examples 1–3] It taught at a rate which it considers as a dicyclopentadiene as a component (A), is considered a 1l. autoclave with styrene and a component (C) as a component (B), and shows an indene in Table 1, and thermal polymerization was carried out to the bottom of nitrogen-gas-atmosphere mind as a solvent using toluene in reaction temperature [of 275 degrees C], and reaction-time 100 minutes. After reaction termination, distillation removed the low polymerization component and a solvent and the resin (I) which has unreacted or the description shown in Table 1 were obtained.

[0027] Then, 2.0g (N-113, JGC make) of nickel system catalysts was taught to the 0.2l. autoclave, having used the cyclohexane as 80g and a catalyst for this resin (I) as 80g and a diluent, and the hydrogenation reaction was performed at G and 300 degrees C 150kg/cm² of hydrogen pressure. After reaction termination, distillation removed the diluent and hydrogenation petroleum resin was obtained. The description of hydrogenation petroleum resin was shown in Table 1.

[0028] The hydrogenation petroleum resin obtained by this invention is excellent in compatibility, and it turns out initial adhesion (tuck), adhesion, holding power, and that it excels also in

economical efficiency further so that clearly from Table 1. Moreover, the hydrogenation petroleum resin of the example especially manufactured under the desirable conditions of a ratio (B/C) was very excellent in the description of all above.

[0029]

[The examples 1-5 of a comparison] Hydrogenation petroleum resin was manufactured using the raw material presentation which is under the same condition as the above-mentioned example, however is shown in Table 1. The description of the obtained hydrogenation petroleum resin was as being shown in Table 1.

[0030] Thus, the hydrogenation petroleum resin obtained by the example of a comparison was fully able to satisfy no binder engine performance as stated above.

[0031]

[Table 1]

表 1

	実 施 例			比 較 例				
	1	2	3	1	2	3	4	5
原料組成、%								
成分 (A)	55	55	55	92	40	55	55	55
成分 (B) + (C)	45	45	45	8	60	45	45	45
(B)/(C)	2.3	4.0	0.7	2.0	0.2	0	0.5	∞
樹脂 (I) 性状								
軟化点、℃	85	87	86	86	70	84	86	83
収率、%	80	81	79	81	79	70	76	82
I ₂ 価, cg/g	215	216	208	235	192	208	205	211
芳香核含量、%	31	32	31	5.6	41	31	32	31
分子量								
Mw	930	1050	850	550	810	530	680	2100
Mw/Mn	1.97	2.13	1.88	1.94	1.82	1.65	1.76	4.11
Mw>5000、%	0.87	1.54	0.45	0.52	0.35	0.33	0.27	9.83
5000>Mw>3000、%	3.05	4.01	2.13	1.45	1.23	0.63	1.07	5.50
水添石油樹脂性状								
軟化点、℃	104	104	103	105	85	102	102	103
I ₂ 価, cg/g	7.7	8.5	8.0	5.0	9.6	9.0	8.8	7.9
芳香核含量、%	19.1	19.5	19.3	1.5	30.8	19.8	20.0	18.9
水添率、%								
エチレン性								
不飽和基	96.4	96.1	96.2	97.9	95.0	95.7	95.7	96.3
芳香核	38.4	39.1	37.7	73.2	24.9	36.1	37.5	39.0
粘着剤性能								
相溶性	◎	○	◎	×	○	◎	◎	×
接着力, g/20mm	1350	1100	1350	300	1200	1550	1400	600
保持力, Hr.	1.5	1.4	1.1	0.5	0.2	0.5	0.8	1.1

The rate of hydrogenation of an ethylene nature partial saturation radical, the rate of hydrogenation of a nucleus, compatibility, adhesive strength, and holding power show the following contents among Table 1.

[0032]

Rate (%) of hydrogenation = (aromatic series content of aromatic series content / petroleum resin before hydrogenation of 1-hydrogenation petroleum resin) x100 compatibility of rate (%) of hydrogenation = (I2 of 1-hydrogenation petroleum resin I2 of ** / petroleum resin before hydrogenation **) x100 nucleus of an ethylene nature partial saturation radical : EVA / petroleum resin / wax Blend with 40/40/20 (weight ratio), and an observation adhesive strength: melting sample is applied to the thickness of 0.1mm for the transparency of the mixed liquor which fully carried out dissolution mixing on an aluminium sheet. A spreading side comrade is pasted up at 160 degrees C using a heat sealer, a measurement holding power: melting sample is applied to corrugated paper adherend at the hauling rate of per minute 30cm with a hauling testing machine, and it sticks on another corrugated paper adherend immediately. Time amount until it applies a 200g load in 45-degree-C oven and a spindle falls this test piece

[Translation done.]

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☒ **BLACK BORDERS**
- ☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☐ **FADED TEXT OR DRAWING**
- ☒ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.